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Exhibit H .

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Reexamination of) and Reissue of: U.S. Patent No. 5,464,551 Issued: November 7, 1995 Inventor: GERBRAND DEETMAN Assignee: Solutia Inc. Reexam Control No.: 90/004,683 Filed: July 2, 1997 Reissue Application No.: 08/966,425 Filed: November 7, 1997 STABILIZED PHOSPHATE ESTER-BASED FUNCTIONAL FLUID COMPOSITIONS)

Assistant Commissioner for Patents Washington, D.C. 20231 Box: Reexamination

sir:

Second Declaration Of Dr. Terry C. Wolfe Under 37 C.F.R. \$1.132

I, Terry C. Wolfe, hereby declare as follows:

1. I am the same Terry C. Wolfe that previously submitted a declaration in connection with the above-identified reexamination application. I understand that my prior d claration was also submitt d as evidence in the aboveidentified reissue application papers. My education and work experience with hydraulic fluids is set forth in that prior

declaration.

- 2. I have been advised that those two proceedings have been merged. I understand that Solutia is submitting this Declaration as part of a response to an Office Action issued August 19, 1998 in connection with the merged proceeding. I am familiar with the claims pending in the reexamination and reissue applications. I have also reviewed and am familiar with the August 19, 1998 Office Action.
- 3. As explained in my prior declaration, the claimed invention concerns flame retardant phosphate ester based functional fluid compositions. The invention is directed to fluids that have improved base stock formulations as well as to fluids that contain the novel additive packages. In my prior declaration, I discussed experiments that had been conducted on several Type IV and Type V functional fluid compositions to investigate the effect on thermal stability of these elements, i.e., the substantially C₄-C₅ isoalkyl substituted phosphate ester base stock and the novel additive package, from both the '551 patent and a more recent set of experiments.
- 4. I understand that the Examiner has reviewed the results of these experiments and questioned whether the effects shown are attributable to the combination of elements, either element alone, or a subcombination of the elements. In order to clarify these prior results, a further set of experiments was conducted on several fluid compositions in order to more clearly demonstrate the effect on thermal stability of (i) the isoalkyl phosphate ester base stock compositions (discussed in

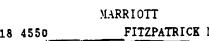
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¶¶ 5-9) and (ii) the novel additive composition (discussed in ¶¶ 10-13}.

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- 5. In order to demonstrate the effect on thermal stability due to the use of a substantially iscalkyl phosphate ester base stock, fluids were prepared as described in the table attached to this Declaration as Exhibit 1.
- 6. As seen from the table the compositions contained the same combination of additives, representative of a Type IV additive package, but differed in that compositions A and B contained tri-normal alkyl phosphate ester base stock ("TBP") and compositions C and D contained a tri-isoalkyl phosphate ester base stock ("TIBP"). The remaining ingredients are defined in Example 12 in the '551 patent. Reclube 110 is also known as Kronitex 100, which is defined in the '551 patent. HF-411, a viscosity index improver disclosed in the '551 patent, comprises 35.5% solids as it is employed both in tributylphosphate esters for compositions A & B and in triisobutylphosphate esters for compositions Compositions A & B were divided in half and the second half of the composition was used in the later experiments discussed at ¶ 10 below. Composition G is the second half of A and Composition H is the second half of B.
- 7. Compositions A-D were each divided into three samples of approximately 45g each for testing. Prior to thermal stability testing, water was added to the samples to adjust their water content to between 0.42 - 0.46% for compositions A and B and between 0.45 - 0.49% for compositions

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These water levels approximate "in service" conditions. The thermal stability testing methods used were the same as explained in my prior declaration and as used in the '551 patent. The following table shows the thermal stability results (Hours at 325°F) comparing TIBP with TBP using same Type 4 additive package:

<u> </u>	BF_based flu	ids TIBP	based fluids
Composition A:	249 ·	Composition C:	352
	280		423
	218		351
Composition B:	300	Composition D:	372
	311		450
	248		382
Average StD	268 35		388 40

- 8. The results of the thermal stability experiments show that fluid compositions containing the isoalkyl base stock compositions have improved thermal stability when compared to the fluids with normal alkyl base stock compositions when using the same Type IV additive package. The improvement is statistically significant based on statistical analysis (t-Test) of the results.
- 9. The results of this experiment, when viewed with the prior evidence in the '551 patent and my first declaration, demonstrates that the benefits of isoalkyl substituted



phosphate ester bas stocks are not tied to any particular additive, but can be attributed to the isoalkyl substituted phosphate ester. In particular, the additive package in the fluids tested in this experiment did not contain an amine antioxidant, but contained only "IONOL", which is a hindered phenol antioxidant, yet showed significantly improved thermal stability when isoalkyl phosphate esters were used.

- 10. In order to demonstrate the effect on thermal stability due to the use of the novel additive package of the invention, additional fluids were prepared as described in the table attached as Exhibit 2.
- disclosed in the '551 patent at Example 12 except for Poly 4495, which is a polyalkyl methacrylate viscosity index improver of the type disclosed in the '551 patent. Similar to HF-411 described above, Poly 4495 comprises 35.5% solids as it is employed in tributylphosphate esters for compositions E and F. As discussed above in § 6, the second half of Compositions A & B were used as Composition G and Composition H, respectively. Thus, all of the fluids contained a tri-normal butyl phosphate ester base stock composition representative of prior art phosphate ester base stock compositions but differed in that Compositions G and H contained an additive package representative of a Type IV fluid and Compositions E and F contained the novel additive package of the invention.
- 12. Compositions E-H were each divided into three samples of approximately 45g each for testing. Prior to

thermal stability testing, samples E and F were determined to contain 0.10 - 0.11% water and samples G and H were determined to contain between 0.11 - 0.12% water. The thermal stability testing methods used were the same as used above in ¶¶ 5-9. The following table shows the thermal stability results (Hours at 325°F) comparing Type IV additive packages with the novel additive package of the invention:

	Type 4	fluids	Type 5	fluids
Composition (G: 739	Composition	E: 907	
	7	705		881
	7	717		838
Composition F	H: 752	Composition	F: 927	
	7	84		836
	7	770		911
Average StD		45		887 42

experiment show that fluids containing the novel additive package of the invention, when added to prior art phosphate ester base stock formulations, have improved thermal stability when compared to the fluids with a Type IV additive package. The improvement is statistically significant based on statistical analysis (t-Test) of the results. It should also be noted that the differences in the times between Compositions A and B in the first experiment and Compositions G and H in the

second experiment can be attributed to the greater amount of water present in Compositions A and B.

All statements made herein of my own knowledge are true and all statements made herein on information and belief are believed to be true. This Declaration is made with the understanding that any willful false statements and the like so made are punishable by fine or imprisonment, or both (18 USC 1001) and may jeopardize the validity of the application or any patent issuing thereon.

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Exhibit 1

Compositions:

	. 6	6620	o -	70.0	77 71	88	2	*	ė	
ત્રુ					y u	9.98		*	67.97	100.
D Wt. g	0.090	, L	3.04	0.0034	17,17	30.05	74, 34		204.59	301.0134
* U	0.023	in 0	0.995			86.6	14.61		68.04	1 100.
Wt., g	0.07	1.49	2.99	0.0031	17.59	30.01	43.91	;	204.53	300.5931 100.
o% ©	0.025	0.5	0.998	0.0011	5.816	96.6	14.59	68.11		6 100. (100.0001)
Wt., 9	0 	2.99	6.01	0.0066	35.04	· E0 * 09	87.93	410.31	}	602.4666 100.
o%	0.025	0.5	1.003	0.0009	5.81	10.0	14.44	68.22		100,
Wt., g	0.15	3.0	6.03	0.0056	34.92	60.09	86.81	409.95	1	600.9556
	FC-9.8	FH-132	IONOL	Dye	MCS1.562	Reolube- 110	HF-411	ľBP	ribp	lotal





Compositions:

	₩1:. q %	Wt. g · s
Van Lube	0.07 0.023	0.09 0.030
FC-98	0.07 0.023	0.07 0.023
IONOL	2.12 0.706	2.10 0.701
E330	1.36 0.453	1.36 0.454
DODPA	1.36 0.453	1.36 0.454
FH-132	1.52 0.506	1.54 0.514
Dye	0.0039 0.0013	0.0032 0.0011
MCS-1562	17.57 5.85	17.53 5.85
S-154	9.14 3.043	8.94 2.983
Poly 4495	38.68 12.88	38.67 12.90
TBP	228.43 76.06	228.0 76.09
Total	300.3239 100. (99.9983)	299.6632 100. (100.0001)